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HPC 70-109

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THE BRITTLE FRACTURE OF AMORPHOUS THERMOPLASTIC POLYMERS

BY

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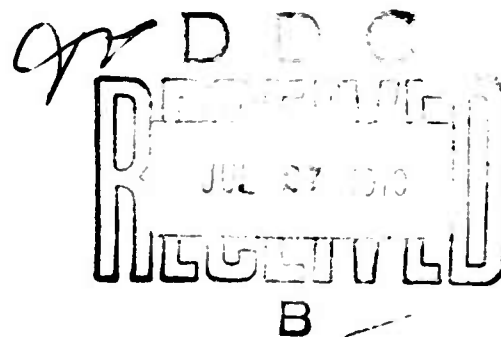
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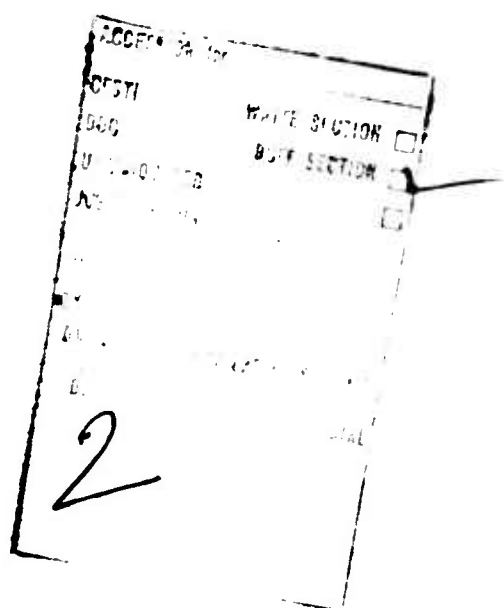
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BY

A. T. DiBENEDETTO
K. L. TRACHTE

FEBRUARY 1970

FOREWORD

The research reported herein was conducted by the
staff of the Monsanto/Washington University Association under
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of Naval Research, N00014-67-C-0218 (formerly N00014-66-C-0045),
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THE BRITTLE FRACTURE OF AMORPHOUS THERMOPLASTIC POLYMERS

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Abstract

The brittle fracture properties of polyphenylene oxide, polysulfone, polycarbonate, and polymethylmethacrylate thermoplastic polymers were investigated over a wide range of temperatures. Fracture energy measurements were made using double edge-notched tensile samples. Tensile strength, tensile strain, and initial elastic modulus were measured for calculation of the fracture energy and further analysis of the polymer behavior.

It was found that mechanical transitions in the tensile properties corresponded reasonably well with transitions in the fracture energy in the temperature range investigated. Fracture surface photographs permitted visual analysis of the fracture process. It was found that the roughest fracture surface corresponded to the maximum in the fracture energy for a given polymer.

A theory for prediction of polymer tensile yield strain is presented, based on the volume dilation concept. The implications of this theory are discussed in terms of the crack tip flow process leading to brittle fracture.

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THE BRITTLE FRACTURE OF AMORPHOUS THERMOPLASTIC POLYMERS

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Introduction

There is substantial evidence in the literature that viscous flow or plastic deformation occurs near the tip of an advancing crack in glassy amorphous polymers. The measured fracture energy for glassy polymers is of the order of 1,000 times greater than the theoretical surface energy of the solid. This increase has been attributed to the energy requirements for viscous flow and polymer crazing at the crack tip.

It is reasonable to expect that, as plastic deformation becomes more inhibited by successive hindrance of the side group and main chain motions of the polymer molecule, the fracture energy should decrease toward the theoretical surface energy, which involves only breaking of atomic bonds.

Unexpectedly, it has been found that for polymethylmethacrylate (Plexiglas II UVA), the fracture energy continues to increase with decreasing temperature, at least to -60°F. A three-fold increase in fracture energy was found as the test temperature was reduced from the main glass temperature, T_g, at 220°F to -60°F. This data is in agreement with previous work [1, 2] on the same grade polymer. This is surprising in that one would expect plastic deformation to be already considerably reduced at this lower temperature, which is about 260°F below the main glass temperature.

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Since it was presumed that a maximum in the fracture energy must occur some-
where between the main glass temperature and absolute zero of temperature, three
amorphous polymers with high main glass temperatures were investigated, allowing a
wide temperature range for testing. The four polymers investigated were:

1. Polyphenylene oxide (General Electric Grade 631-111)

$$T_g = 410°F$$

2. Polysulfone (Union Carbide Grade 1700)

$$T_g = 375°F$$
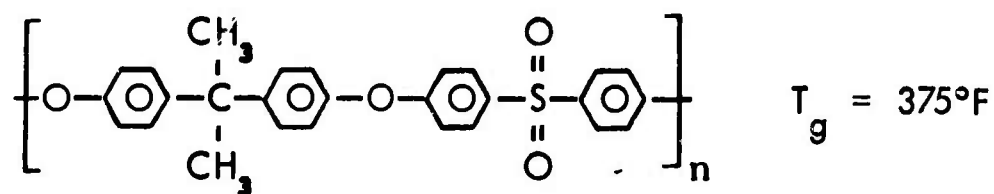
3. Polycarbonate (General Electric "Lefan")

$$T_g = 290°F$$
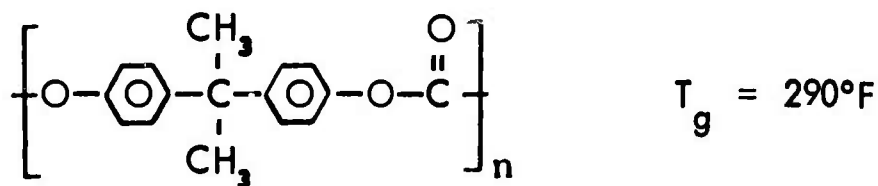
4. Polymethylmethacrylate (Rohm and Haas Plexiglas II UVA)

$$T_g = 220°F$$

Theory

The fracture mechanics of Irwin [3] was used to calculate the plane strain frac-
ture energy, γ, required for catastrophic failure of the polymers. The sample geometry

used is shown in Figure 1. An iterative technique utilizing a computer was used to solve Irwin's approximation for the chosen geometry:

$$K_{I_c} \simeq \sigma_n \left\{ W \left[\tan \frac{\pi (a + K_{I_c}^2 / 2\pi \sigma_y^2)}{W} + .1 \sin \frac{2\pi (a + K_{I_c}^2 / 2\pi \sigma_y^2)}{W} \right] \right\}^{1/2} \qquad (1)$$

where K_{I_c} = critical stress intensity parameter at the crack tip; σ_n = brittle tensile strength of a notched sample, based on the gross cross-sectional area; W = sample width; a = half-crack length at fracture; σ_y = tensile yield strength at the notch tip. The plane strain fracture energy γ was calculated from:

$$\gamma = K_{I_c}^2 (1 - v^2) \, 2E \qquad (2)$$

where v = Poisson's ratio of the polymer (taken as .35 for the four polymers); and E = initial elastic modulus of the polymer.

From the notched sample, the experimental values of W, σ_n, and a were obtained. The brittle tensile strength, σ_n, was calculated directly from the load-deformation curve for the sample. The value of a, which includes the initial machined notch length and the slow crack growth prior to failure, was measured from the fractured sample surface to the nearest $\pm .003$ cm under a microscope.

The values of σ_y used in the calculation of γ were obtained from a standard ASTM Tensile test D1708 [4]. A three-inch gauge length was used with all other dimensions as specified by ASTM. The strain rates used in our testing program were .013/min for the tensile tests and .25/min for the notched sample tests. The strain rate at the

crack tip, however, is likely to be much higher than that of the applied strain rate [5]. Since polymer properties are strain rate sensitive, the value of σ_y obtained from the tensile test is expected to be lower than the actual yield strength at the crack tip. However, an error analysis based on Equation (1) indicates that this uncertainty in σ_y does not significantly affect the calculated fracture energy within a three-decade range of strain rates [6].

The values of initial elastic modulus, E, used in the calculations were those obtained from the unnotched tensile tests.

The test temperature was varied from -60°F to the main glass transition temperature of the polymer. The sample preparation and testing procedures have been discussed in more detail elsewhere [6, 7].

Experimental Results

The effect of temperature on the energy required for polymer brittle fracture is shown in Figure 2. A maximum in γ was observed for polyphenylene oxide and polysulfone about 150°F below the main glass transition temperature. Our testing procedure did not allow for thick enough samples of polycarbonate to be tested to keep the fracture in a plane strain mode; consequently, some macroscopic yielding was observed around the fracture plane above 80°F. This is shown in Figure 3. Gross yielding at the notched section resulted in high values of γ, which are not reported with the plane strain data. The plane strain fracture data obtained for polycarbonate are similar to that of polyphenylene oxide and polysulfone.

The degree of roughness of the fracture surface is a qualitative measure of the fracture energy. For instance, the maxima in fracture energy for polyphenylene oxide and polysulfone occur at 250°F and 220°F, respectively. This is also the temperature region of maximum roughness, as seen from Figures 4 and 5. Polymethylmethacrylate exhibits a continuing increase in γ with decreasing temperature, with a corresponding increase in surface roughness as can be seen in Figure 6. As the test temperature approached the main glass temperature (220°F) for this polymer, a near perfect cleavage surface was formed (219°F). A temperature increase of only a few degrees completely changed the fracture mode. At 221°F, gross yielding occurred, resulting in shear failure at the notched section. This same phenomenon was observed with polysulfone and polyphenylene oxide just above T_g.

Over the past few years there have been numerous attempts to relate impact strength to secondary glass transitions. Perhaps the best survey to date is that of Boyer [8]. A comparison of the observed temperature response of γ with known secondary transitions for the four polymers [9-13], showed no apparent correlation between fracture energy and secondary transitions. In fact, the data did not reflect any change in γ at any of the secondary glass transitions.

For this reason, we concluded that the maximum in the plane strain fracture energy for polysulfone, polyphenylene oxide, and presumably polycarbonate is a manifestation of the approaching main glass temperature. This is supported by the tensile yield strength, yield strain, and initial elastic modulus data for the four polymers, as shown in Figures 7, 8 and 9. As the test temperature is lowered from the main glass

transition, the fracture energy increases for all four polymers. The tensile yield strength, yield strain, and initial elastic modulus also increase, at the same rate for all four polymers. Between T_g - 50°F and T_g - 100°F, the rate of increase in the tensile properties decreases abruptly for polyphenylene oxide, polysulfone, and polycarbonate. Considering the difference in strain rates between the tensile and notched tests, this mechanical transition compares reasonably well with the temperature, T_g - 150°F, at which the maximum in γ is observed for polyphenylene oxide and polysulfone (and presumably polycarbonate in plane strain fracture mode). The tensile properties of Plexiglas II UVA increase linearly throughout the temperature region investigated, with no change in slope with temperature. Similarly, γ for Plexiglas II UVA continues to increase as the test temperature is reduced.

According to the above interpretation, the maximum and consequent decreases in γ as T_g is approached is the result of increased polymer softening, as indicated by the tensile data.

Analysis of Data

The mechanism causing the onset of polymer flow has been a subject for speculation for considerable time. There is reason to believe that temperature rise, volume dilation, and a number of molecular factors such as chain entanglement, backbone flexibility, side group size, etc. are involved in the plastic deformation process. There has been considerable support in the literature for the concept of volume dilation as at least a partial cause of yielding in polymers [14-21]. Our tensile yield data appears to further support this concept. The volume dilation theory suggests that as a uniaxial stress is

applied to the polymer, the volume increases according to the relation:

$$\Delta v = (1 - 2v) v \epsilon \qquad (3)$$

where Δv = increase in polymer volume, v = volume of the polymer in the unstressed state, v = Poisson's ratio and ϵ = uniaxial tensile strain.

If part or all of this volume increase corresponds to polymer free volume generation, then yielding will occur when the free volume generated at the test temperature equals that needed for the polymer molecules to "feel" as though they are at the main glass temperature. This yield process is then dependent on what is referred to as a "pseudo" reduction of the main glass temperature, i.e. $\Delta T_g = T_g - T_{test}$.

There have been at least two theories developed which propose that all of the volume increase from an applied load becomes free volume [16, 18]. Although our yield strain data can be fitted to portions of each of the proposed theories, neither theory successfully predicts the yield strain over the entire temperature range tested.

We propose that not all of the increase in volume during loading becomes free volume, but rather that the dilatation associated with the initial linear elastic response of the polymer does not result in free volume. The total strain to yield is expressed as the sum of two terms, a linear elastic strain, ϵ_e, and a non-linear strain, ϵ_v, which for lack of a better name will be called a viscous strain. This is shown schematically in Figure 10.

The elastic component is given by σ_y/E where E is the initial elastic modulus. This is the strain which would be present in the polymer at the yield stress level if no

non-linear deformation occurred. One might visualize this to be the strain in the material if there were no change in the conformation or orientation of the molecules in the glassy state. Rather, the dilation occurs, as it does in simple crystals, with essentially no change in molecular conformation other than an increase in the average spacing between molecules. It is assumed that this elastic deformation does not generate additional free volume.

All of the free volume needed to cause gross yielding is assumed to come from the non-linear increment ϵ_v to the total strain. It is emphasized that this viscous flow model represents plastic deformation on a micro level, perhaps the same as in craze formation. It should not be confused with the post-yield homogeneous plastic flow that occurs during cold drawing of a polymer. Thus, the non-linear incremental strain results in a molecular rearrangement that causes an increase in the free volume of the polymer. This latter effect may or may not be accompanied by an additional macroscopic volume change. The relationship between ϵ_v and the free volume is shown with the aid of Figure 11. Our definition of free volume is identical to that used by Litt and Tobolsky [16]:

$$\Delta v_{free} = \Delta v_a - \Delta v_c = \alpha_{ga} \Delta T_g v_a - \alpha_{gc} \Delta T_g v_c \qquad (4)$$

where Δv_{free} = change in specific free volume (cc/g), Δv_a = change in amorphous specific volume (cc/g), Δv_c = change in close-packed specific volume (cc/g), α_{ga} = volumetric coefficient of thermal expansion in the amorphous glassy state (1/°F), α_{gc} = volumetric coefficient of thermal expansion in the close-packed glassy state (1/°F), v_a = amorphous specific volume (cc/g), v_c = close-packed specific volume (cc/g), and $\Delta T_g = T_g - T_{test}$ (°F).

It should be emphasized that although the polymers investigated are highly amorphous, under certain conditions a close-packed or crystalline-like structure can be induced. For instance, the close-packed specific volume of polycarbonate [17] was taken as that value of specific volume obtained from x-ray scattering data on well-annealed polymer samples.

Incorporation of Equation (4) into Equation (3) results in the expression:

$$\epsilon_v = \frac{\Delta v_{free}}{v_a (1-2v)} = (\alpha_{ga} - \alpha_{gc} \frac{v_c}{v_a}) \Delta T_g / (1-2v) \tag{5}$$

Thus the yield strain for the polymer is given by:

$$\epsilon_y = \sigma_y/E + (\alpha_{ga} - \alpha_{gc} \frac{v_c}{v_a}) \Delta T_g / (1-2v) \tag{6}$$

The elastic component of the yield strain appears to be the same for all four polymers, as shown in Figure 12. Whether this is true for all amorphous polymers is not known, but it is not too likely. Subtraction of the experimental elastic strain in Figure 12 from the experimental yield strain in Figure 8 results in the viscous strain, ϵ_v, for the four polymers. As shown in Figure 13, except for the initial 50°F below T_g, the data appears to be linear with ΔT_g. Since at T_g no viscous strain is needed to cause yielding, the data lines were drawn through zero at T_g. In accordance with Equation (5), the slope of the line is equal to $(\alpha_{ga} - \alpha_{gc} v_c/v_a)/(1-2v)$. Table 1 shows the values of $(\alpha_{ga} - \alpha_{gc} v_c/v_a)$ for the four polymers as determined from Figure 13. The range of values between 2 to 7 x 10^{-5} (1/°F) appears to be reasonable. The only data available to check the theoretical slope was for polycarbonate [17] and, as seen, the

agreement is excellent. Specific volume measurements on the close-packed state were found at room temperature for polyphenylene oxide and polymethylmethacrylate [16], but a value of α_{gc} could not be found or reasonably calculated to check the theory for either polymer.

Within 50°F of the main glass transition temperature there appears to be a small deviation in the linearity between ϵ_v and temperature, with the experimentally determined values being slightly higher than the linearly extrapolated values. If one includes a temperature dependence in v, α_{ga}, and α_{gc}, the value of viscous strain would be increased in the region near T_g, further improving the fit. In particular, α_{ga} for polymethylmethacrylate has been shown to increase rapidly above 200°F [22]. Poisson's ratio would also be expected to be increasing toward a value of .5 in this temperature region. A more correct form for viscous strain is then:

$$\epsilon_v = \left[\alpha_{ga}(T) - \alpha_{gc}(T)\ \frac{v_c(T)}{v_a(T)}\right] \Delta T_g / [1 - v(T)] \tag{7}$$

Although the yield strain data at these temperatures is not sufficiently accurate to merit calculations of this type, Equation (7) will undoubtedly give a better fit of the experimental data.

Conclusions

The fracture energy of polyphenylene oxide, polysulfone and polycarbonate increases with decreasing temperature to a maximum at roughly 150°F below the main glass transition temperature of the polymers. The maximum appears to be associated with the general changes in viscoelastic properties accompanying the main glass

transition temperature and can be correlated with changes in strength and modulus in this temperature region. A similar maximum in γ with corresponding changes in strength and modulus was not observed for polymethylmethacrylate to as low a temperature as we could study. Presumably polymethylmethacrylate must also show the same type of behavior, but at a much lower temperature than the other three polymers. Below this maximum, the fracture energy decreases almost linearly with decreasing temperature. If one extrapolates the data for three of the polymers to the temperature region of absolute zero, the fracture energy appears to have decreased to the order of the theoretical surface energy.

In addition, the volume dilation theory has been further supported by our polymer yeild strain data. Although no definite mathematical analysis involving strain at the crack tip has been developed as yet, in light of the correlation of the transitions in the fracture energy with the tensile data, the same equations which describe the onset of yielding in a tensile sample may also be applicable to the crack tip yielding process. If this is true, further work of this nature should provide a method for predicting the fracture energy from tensile stress-strain properties.

Acknowledgment

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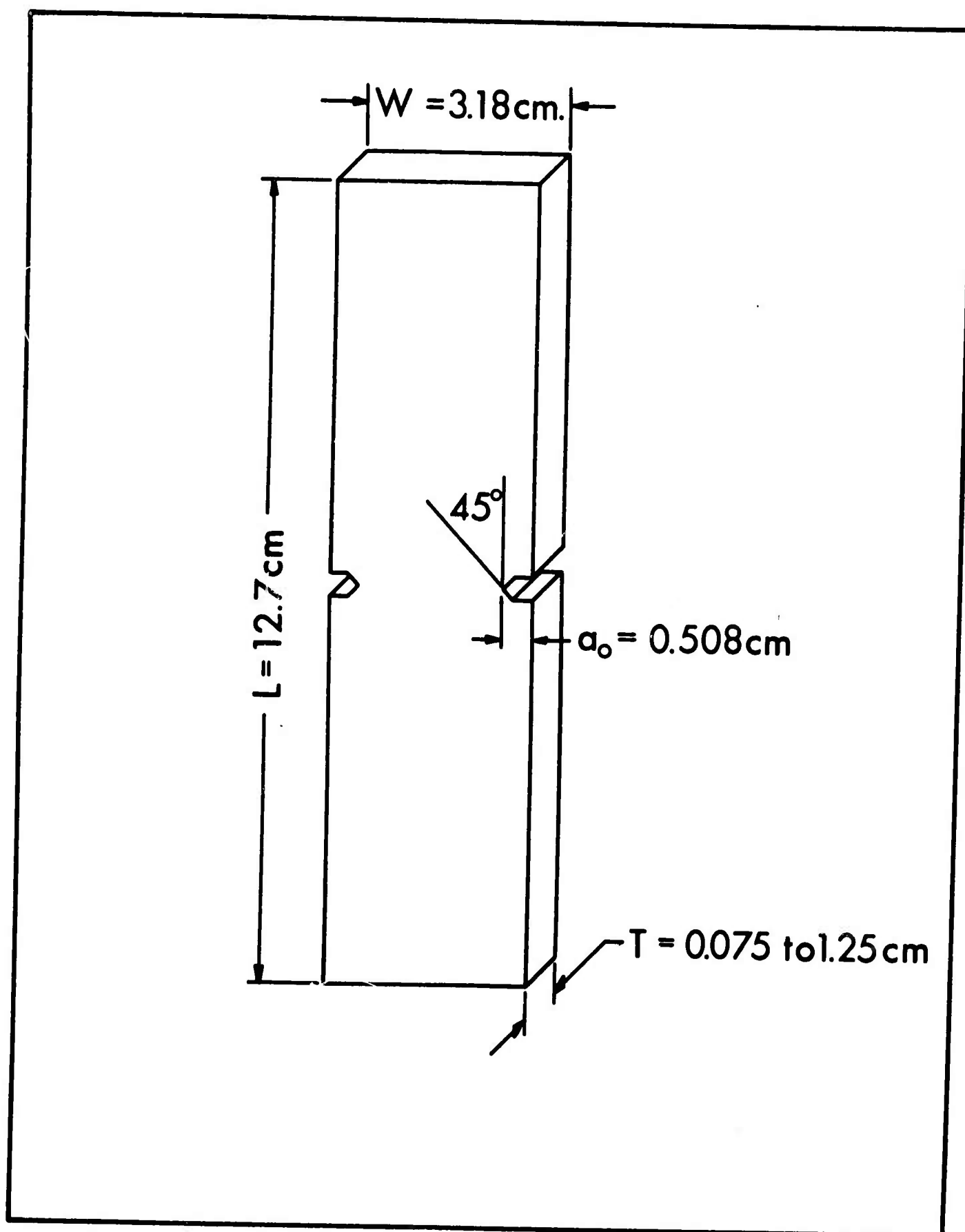
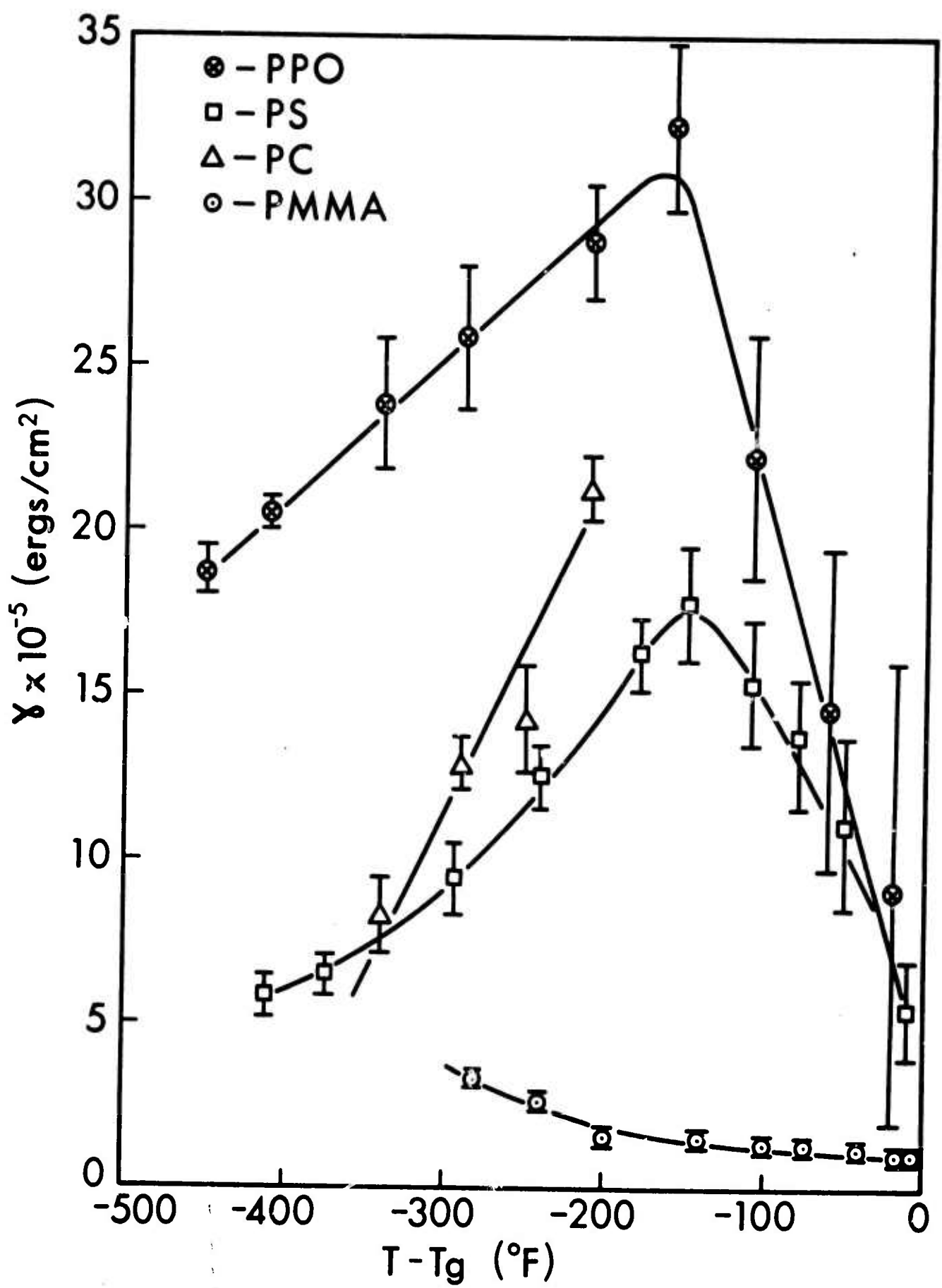Figure 1. SAMPLE GEOMETRY FOR FRACTURE ENERGY TEST

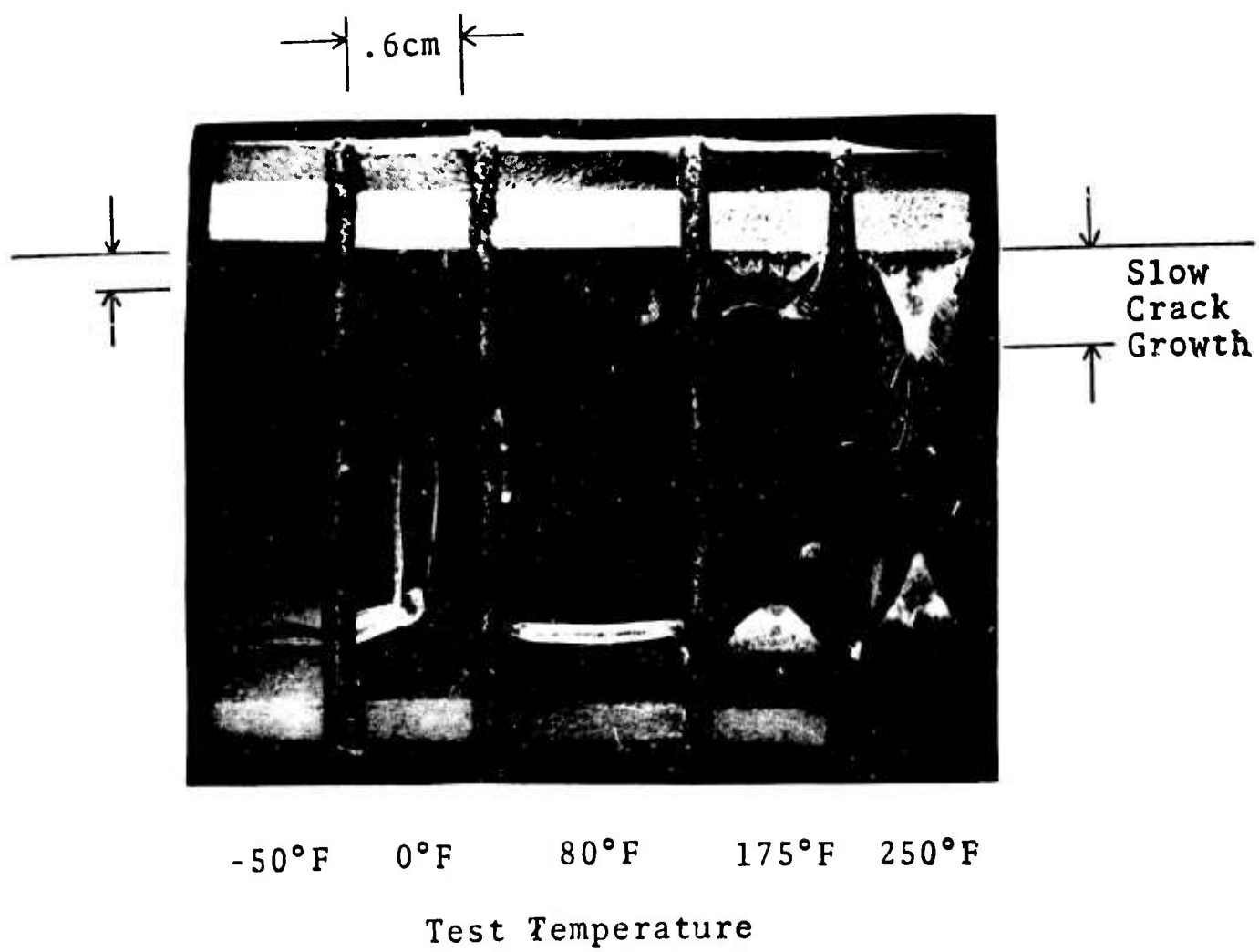Figure 2.   POLYMER FRACTURE ENERGY AS A FUNCTION OF TEMPERATURE

.6cm

Slow
Crack
Growth

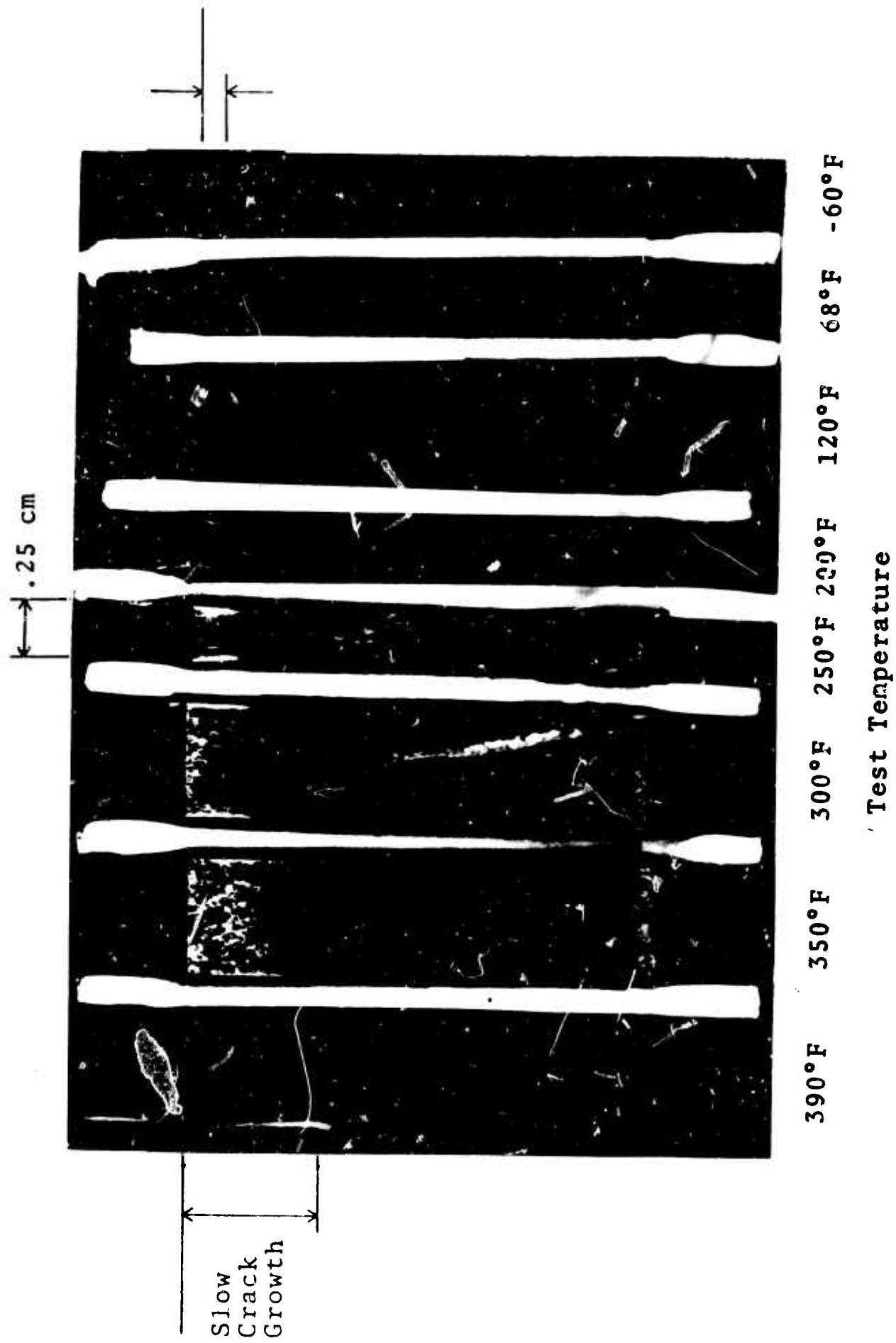-50°F   0°F   80°F   175°F   250°F

Test Temperature

FIGURE 3   POLYCARBONATE FRACTURE SURFACES

.25 cm

Slow
Crack
Growth

390°F   350°F   300°F   250°F   200°F   120°F   68°F   -60°F

Test Temperature

FIGURE 4   POLYPHENYLENE OXIDE FRACTURE SURFACES

Slow
Crack
Growth

.3 cm

-50°F   80°F   135°F   220°F   325°F   365°F
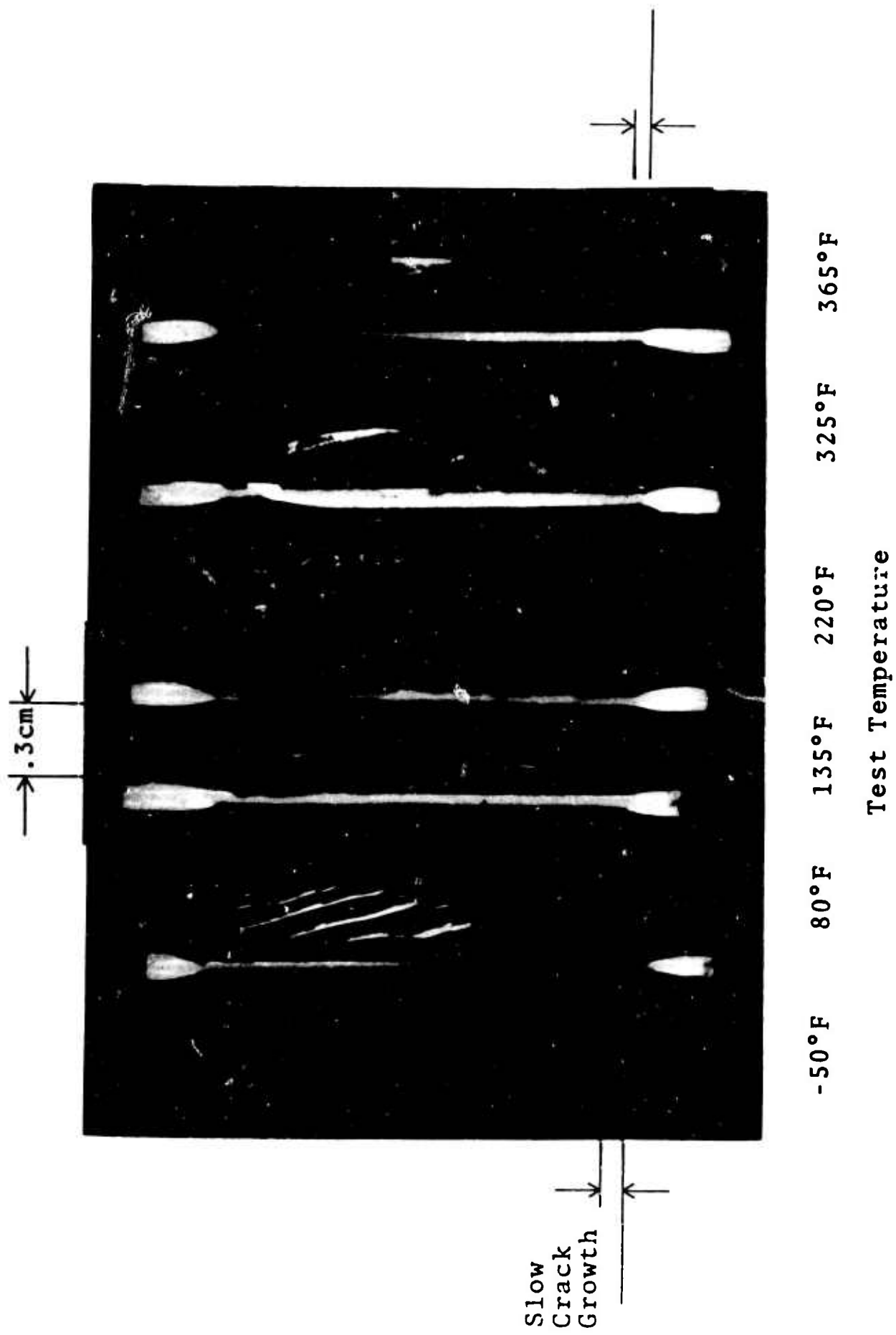
Test Temperature

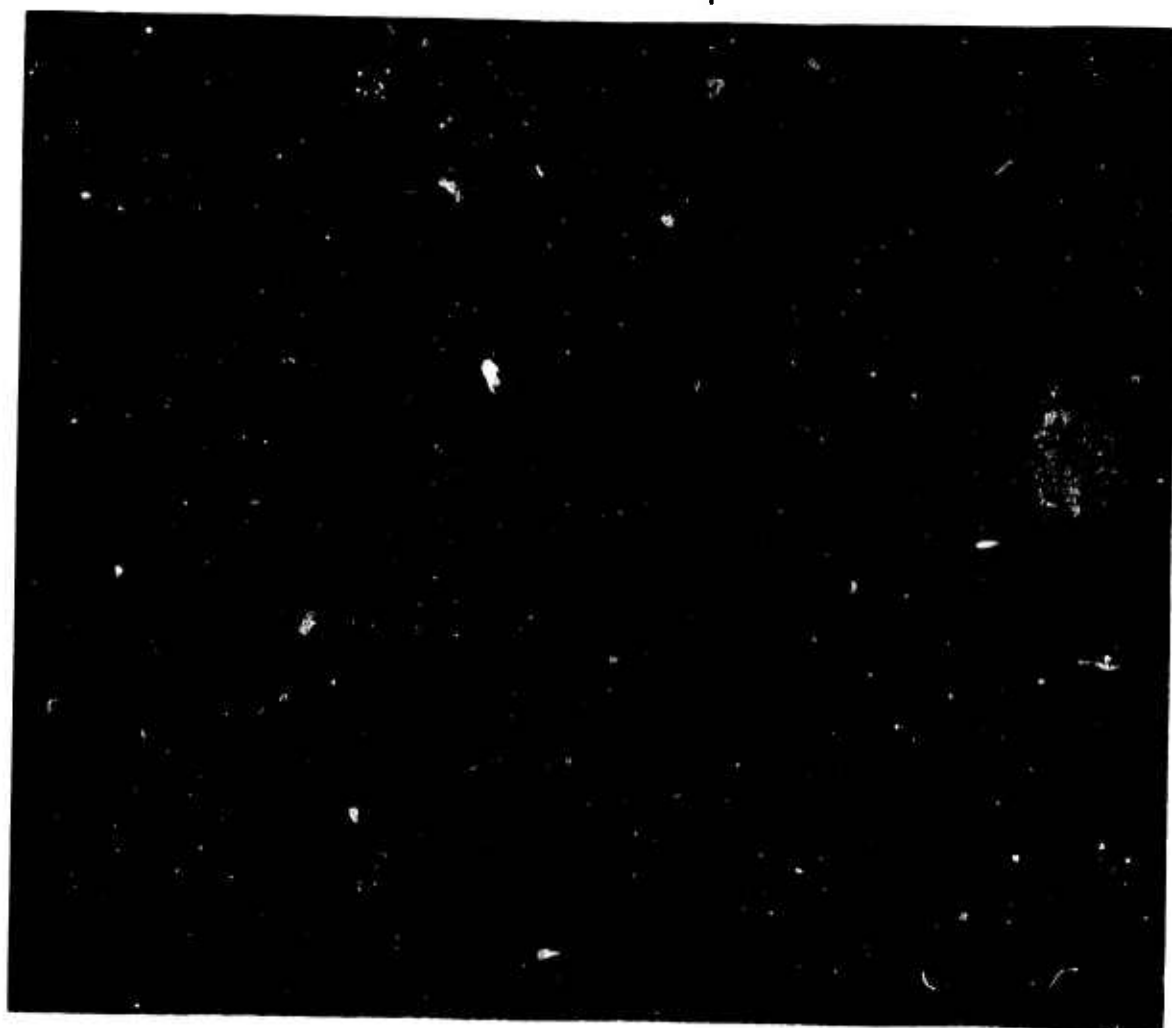FIGURE 5   POLYSULFONE FRACTURE SURFACES

Negligible Slow Crack Growth

→ .63 cm ←

150°F   200°F   215°F   219°F   221°F

Test Temperature

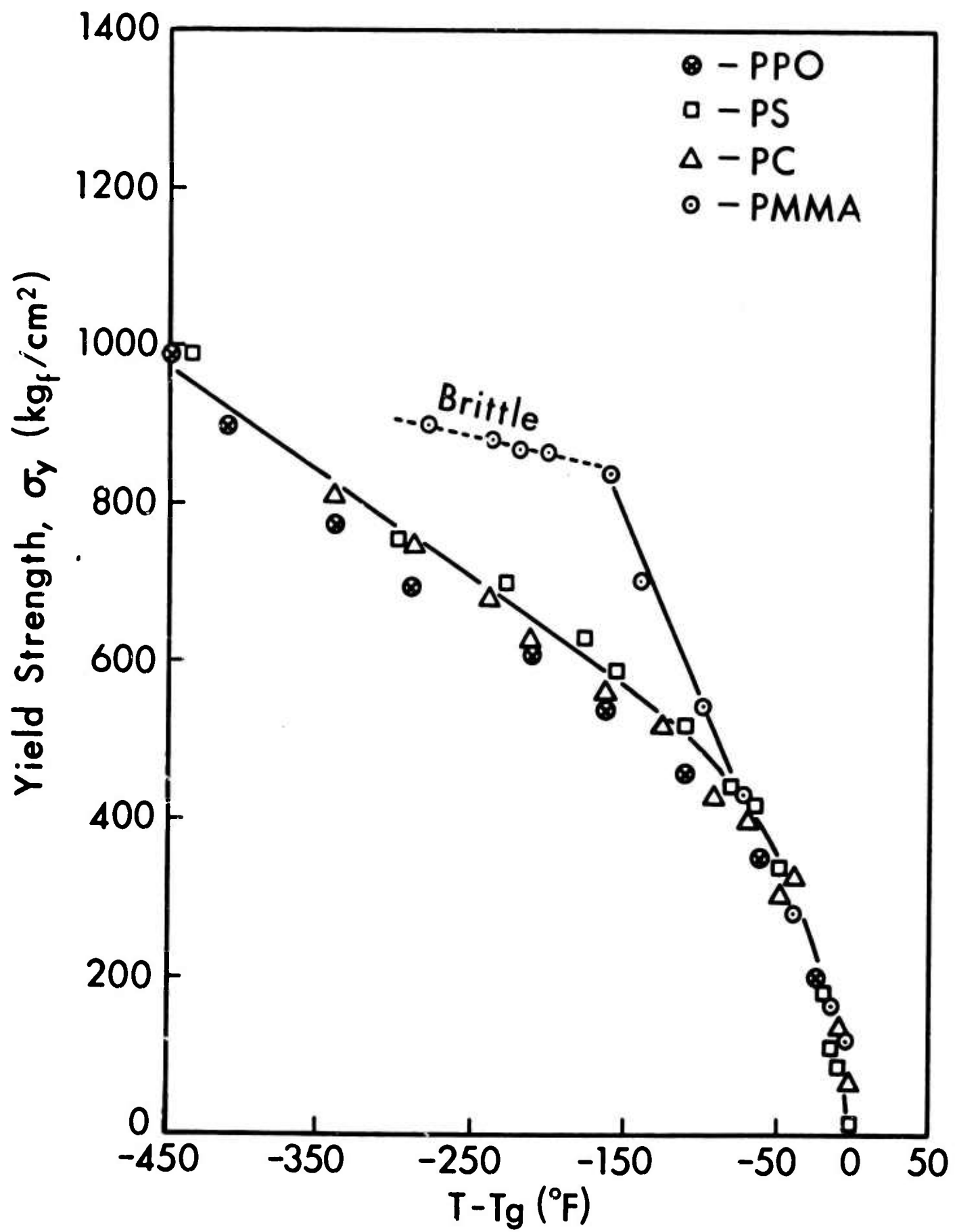FIGURE 6   POLYMETHYLMETHACRYLATE FRACTURE SURFACES

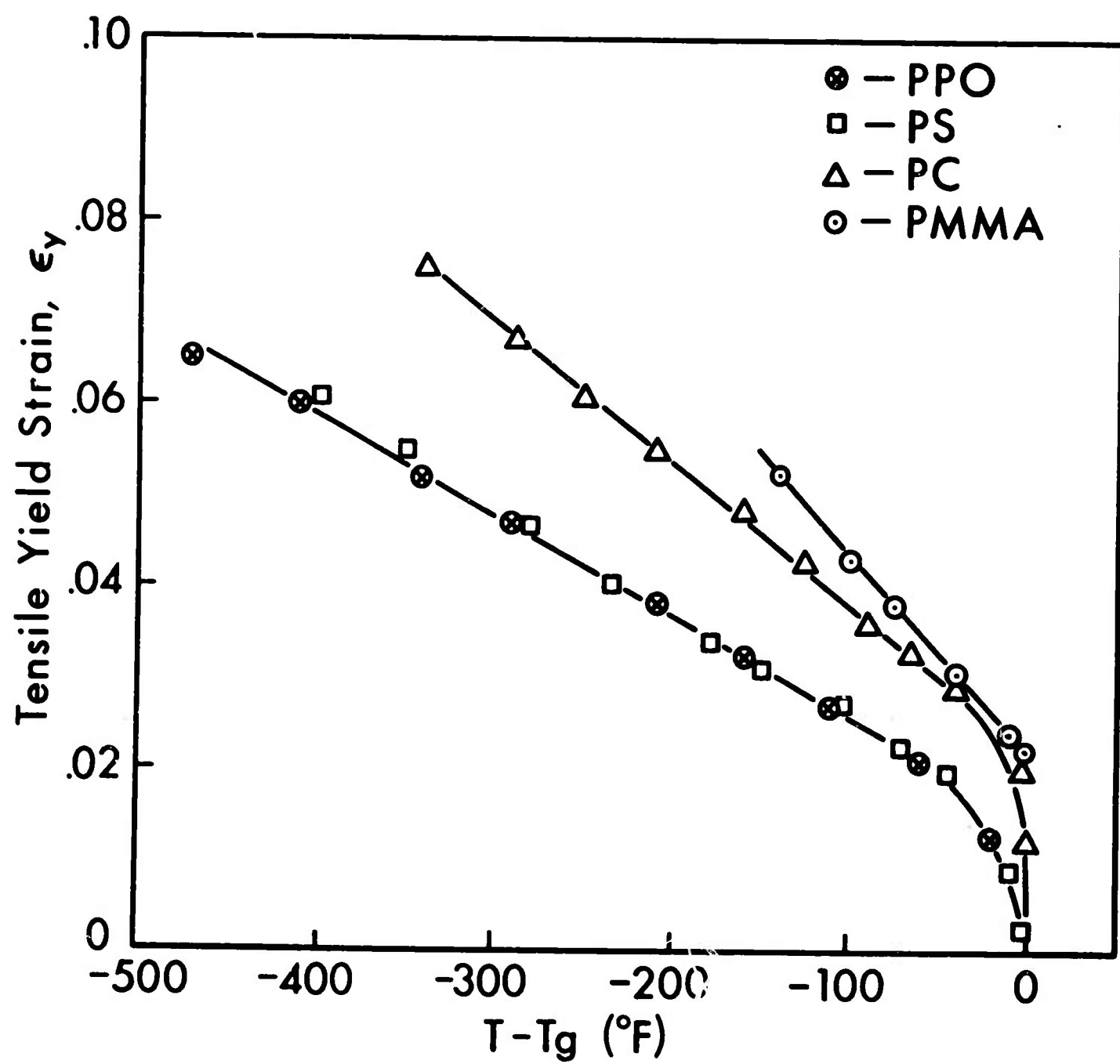Figure 7. EFFECT OF TEMPERATURE ON POLYMER YIELD STRENGTH

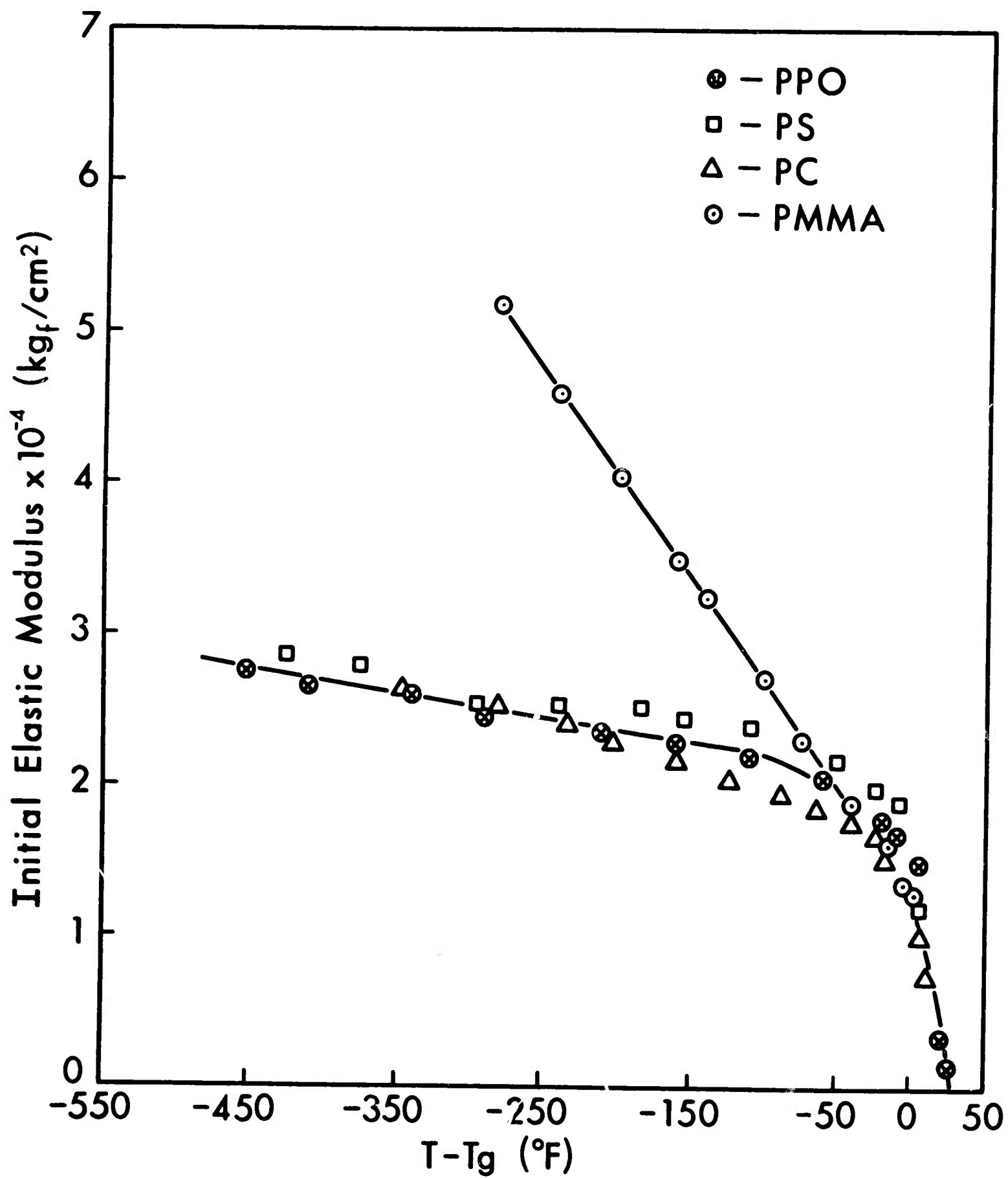Figure 8. EFFECT OF TEMPERATURE ON POLYMER YIELD STRAIN

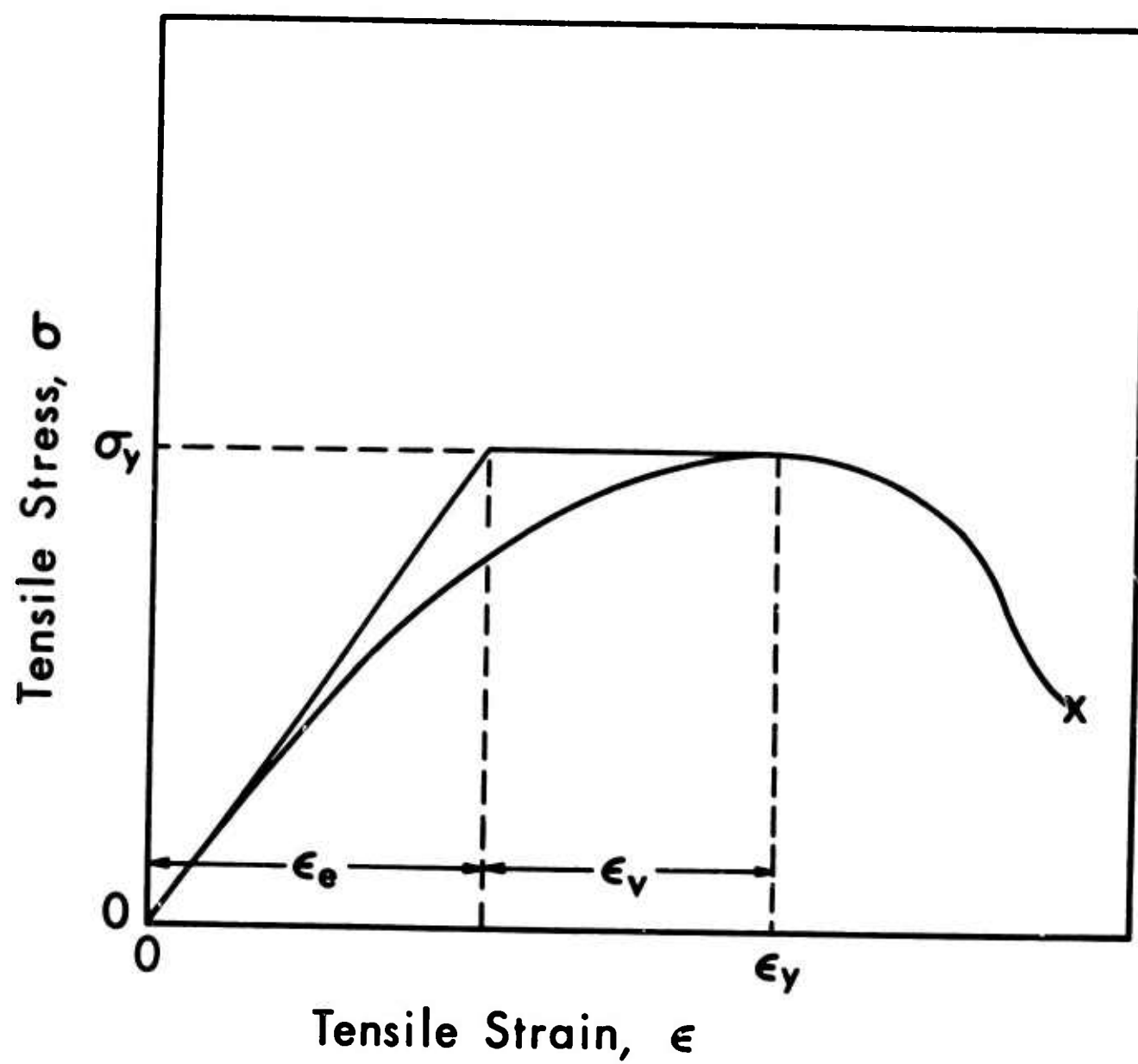Figure 9. EFFECT OF TEMPERATURE ON POLYMER INITIAL MODULUS

Figure 10. SCHEMATIC DIAGRAM OF THE PROPOSED ELASTIC
AND VISCOUS STRAINS

$$\Delta v_a = \alpha_{g_a} \Delta T_g v_a$$

$$\Delta v_c = \alpha_{g_c} \Delta T_g v_c$$

$$\Delta v_{free} = \Delta v_a - \Delta v_c$$

$$\Delta v_{free} = (\alpha_{g_a} v_a - \alpha_{g_c} v_c) \Delta T_g$$

Where $\alpha_g [=] (1/°F)$

Specific Volume, v (cc/gm)

v_a

v_c

Δv_a

Δv_c

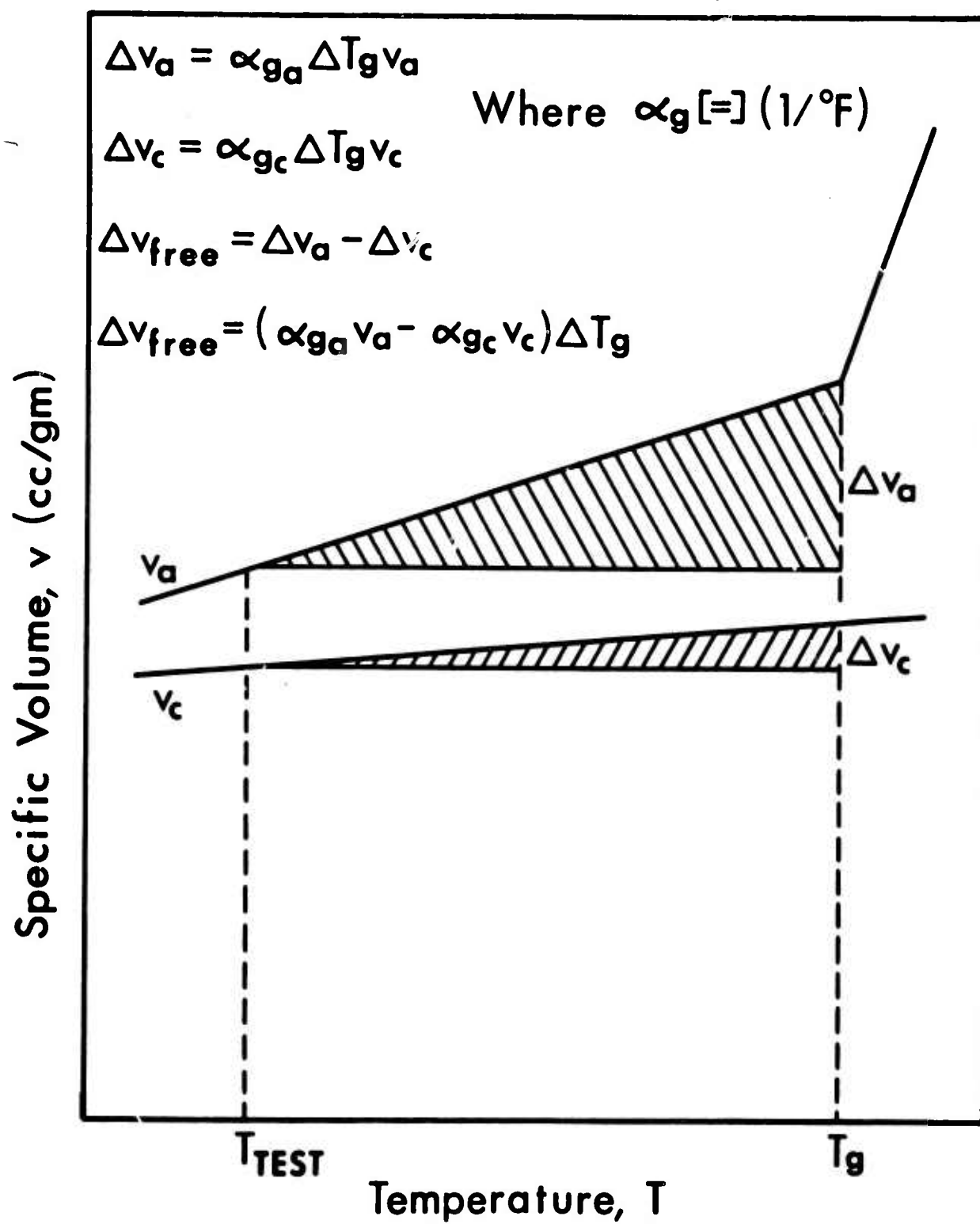T_{TEST}

T_g

Temperature, T

Figure 11. POLYMER FREE VOLUME DEFINED TO INCLUDE
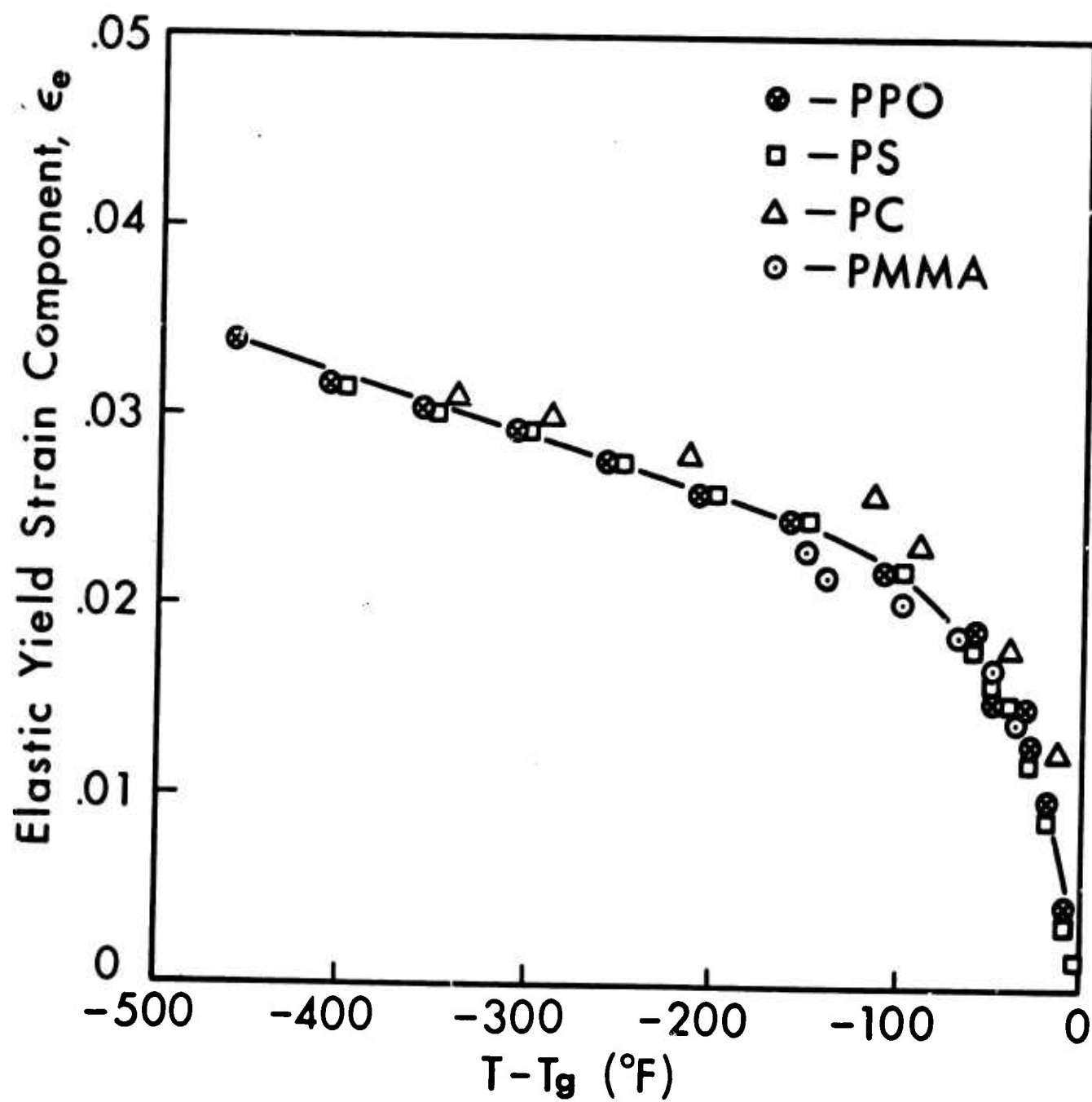CHANGES IN AMORPHOUS AND CLOSE-PACKED
VOLUME

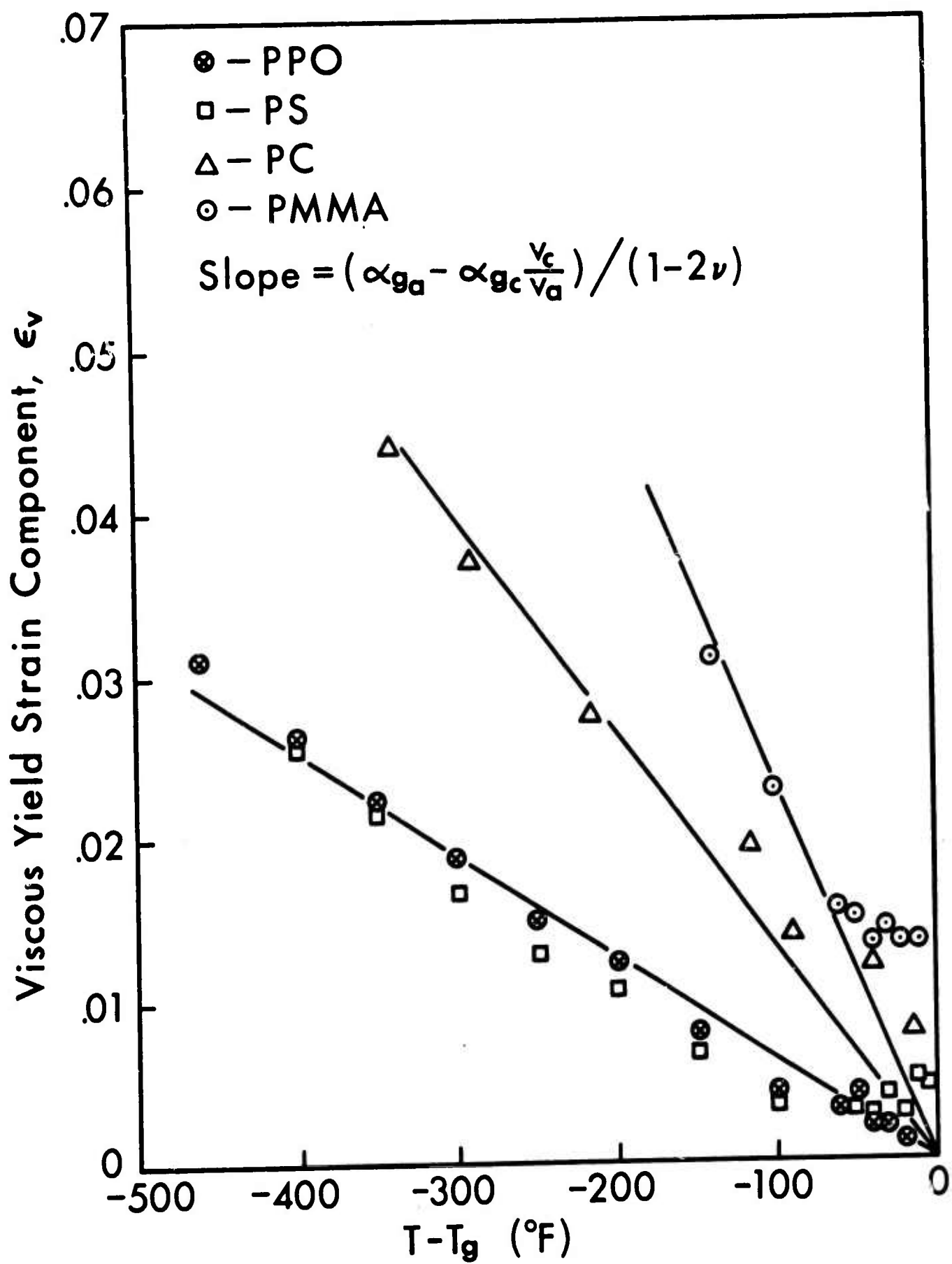Figure 12. VARIATION IN THE ELASTIC STRAIN WITH TEMPERATURE

Figure 13. VARIATION IN THE VISCOUS STRAIN WITH TEMPERATURE

Table 1. COMPARISON OF THE PREDICTED AND MEASURED VALUES OF $\left(\alpha_{ga} - \sigma_{gc}\dfrac{v_c}{v_a}\right)$

Polymer	From Figure 13 $\alpha_{ga} - \alpha_{gc}\dfrac{v_c}{v_a}$ (1/°F)	Measured Value $\alpha_{ga} - \alpha_{gc}\dfrac{v_c}{v_a}$ (1/°F)
PPO	2×10^{-5}	—
PS	2×10^{-5}	—
PC	3.4×10^{-5}	3.6×10^{-5} (16)
PMMA	7×10^{-5}	—

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13. ABSTRACT

The brittle fracture properties of polyphenylene oxide, polysulfone, polycarbonate, and poly-methylmethacrylate thermoplastic polymers were investigated over a wide range of temperatures. Fracture energy measurements were made using double edge-notched tensile samples. Tensile strength, tensile strain, and initial elastic modulus were measured for calculation of the fracture energy and further analysis of the polymer behavior.

It was found that mechanical transitions in the tensile properties corresponded reasonably well with transitions in the fracture energy in the temperature range investigated. Fracture surface photographs permitted visual analysis of the fracture process. It was found that the roughest fracture surface corresponded to the maximum in the fracture energy for a given polymer.

A theory for prediction of polymer tensile yield strain is presented, based on the volume dilation concept. The implications of this theory are discussed in terms of the crack tip flow process leading to brittle fracture.

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